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METHOD AND DEVICE FOR REUSE OF DIMETHYL TEREPHTHALATE SUBLIMATE

[Verfahren und Vorrichtung zum Wiedereinsatz von Dimethylterephthalat-Sublimat]

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Field of invention /1*

The invention concerns a method and a device for separation and recovery of dimethyl terephthalate in the production of polyethylene terephthalate (PETP).

Description of known technical solutions

In the currently practiced methods for production of PETP, the dimethyl terephthalate (DMT) is melted out, heated to the desired temperature in preheaters and intermediately stored. The individual system of vessels are fogged with an inert gas, for example nitrogen, to avoid oxidative decomposition. DMT sublimate is carried by the inert gas escaping through the ventilation line and discharged into the atmosphere or the DMT sublimate is deposited in the corresponding vessels ("Polyester fibers," H. Ludewig, Academie Publishers, 1975).

It is disadvantageous that the DMT sublimate is lost to the production process, and moreover, pollution of the environment results if it is directly discharged in the atmosphere. If the DMT sublimate is deposited in appropriate vessels, there is not a possibility for its reuse, since due to distillation and precipitates of residual moisture components contained in the DMT, the DMT sublimate tends to clump and form encrustations and thus impedes conditions for transport and storage. Measurements showed that the DMT losses can be as much as 0.2% with respect to the amount of DMT that is used. /2 Independent from the high material losses that arise, there are required expenditures to remedy the deposited DMT sublimate for purposes of environmental safety. In addition, if the sublimate ventilation lines go directly to the atmosphere, there is a very high consumption of inert gas.

* [Numbers in right margin indicate pagination of the original text.]

Goal of invention

The goal of the invention is to develop a method and device for deposition and recovery of DMT sublimate in polyethylene terephthalate production.

Nature of the invention

The invention is based on the task of developing a method and apparatus that ensure complete reuse of DMT sublimate at low equipment and manual cost and can be used and transferred to existing polyester plants. It was found that the sublimate that accumulates in the melting or intermediate storage of DMT precipitates in a DMT sublimate separator provided with a glycol trap and is sent back to the production process. The DMT sublimate separator is operated in the form that a pressure > 1000 Pa is generated in the middle of three chambers by the inert gas stream and the fresh glycol needed for the transesterification is passed through the sublimate separator and charged to the reaction system together with the DMT sublimate in solution or suspension. The glycol used as barrier liquid has a temperature of 20-160°C, preferably 110-130°C. The volatile fractions entrained with the inert gas stream are precipitated in a water separator.

The inert gas stream fed to the sublimate separator is used at the same time for befogging the DMT and glycol systems. The fresh glycol pumped through the sublimate separator has a temperature of 70-120°C.

The sublimate separator 3 consists of three chambers 4, 5 and 6, where chamber 5 is provided with, /3
in some cases, a heated ventilation line 13 and a connected water separator 9, chamber 4 is provided with a glycol dispensing line 12 with distribution plate 7, an inert gas dispensing line 11 and a heated DMT sublimate line 10, and chamber 6 is provided with a mixture discharge line 14.

Embodiment example /4

Example 1 (comparison example corresponding to prior art)

The molten DMT that is to be sent to the transesterification is intermediately stored in a preheating vessel. The preheating temperature for the DMT is 170°C. To avoid thermooxidative damage to the molten DMT, the preheating vessel is fogged with an inert gas stream.

Since at a melt temperature over 160°C there is already a high tendency of the DMT to sublime, a considerable amount of DMT sublimate is lost through the ventilation line. At a melt temperature of 170°C and with an inert gas flow of 2 m^3/h or when emptying the preheating vessel at 9 m^3/h, a sublimate amount of 0.2% with respect to the amount of DMT used develops.

Example 2

To avoid the relatively high losses of DMT, the heated DMT sublimate line 10 of the DMT preheating vessel 2 is connected to a DMT sublimate separator 3. For complete separation of the DMT, the DMT sublimate separator 3, which is filled with glycol as barrier liquid, is divided into three chamber systems, where the glycol feed line 12 is installed in the middle chamber 4. The inert gas stream introduced into the same chamber 4 via the inert gas dispensing line 11 has a pressure of 1500 Pa. For ongoing and complete flushing of the DMT sublimate at the chamber walls, the fresh glycol stream is guided over a distribution plate 7.

The DMT sublimate that is in suspension or in solution is diverted to the reaction system in the charging operation, with the glycol being carried away directly via the mixture discharge line 14. The glycol preheater 1 is connected to the DMT system via the ventilation line 13 and water separator 9 that are in chamber 5 of the DMT sublimate separator 3, so that an additional inert gas fogging of the glycol container 1 [sic] can develop.

Through the heating of the glycol barrier liquid in the DMT sublimate separator 3 to a temperature of 110°C, the water that escapes from the preheating vessel 2 with the DMT sublimate is precipitated in water separator 9. /5

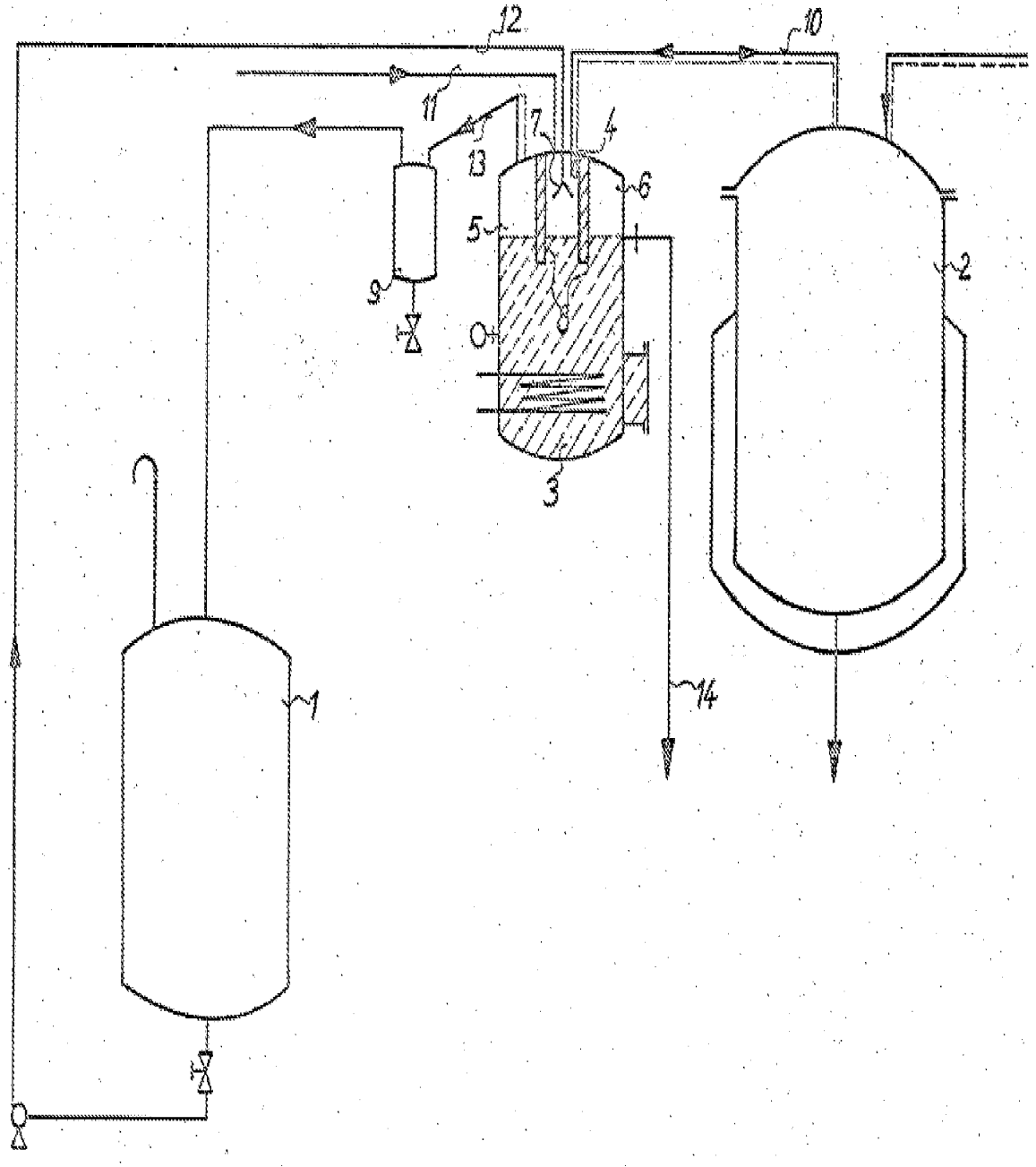
Claims /6

1. A method for reuse of dimethyl terephthalate (DMT) sublimate in the production of polyethylene terephthalate, characterized by the fact that the sublimate that accumulates in the melting or intermediate storage of the DMT is precipitated in a DMT sublimate separator that is provided with a glycol trap and the DMT sublimate separator is operated in the form that a pressure > 1000 Pa is generated in the middle of three chambers by the inert gas stream, the fresh glycol necessary for the transesterification is conducted through the DMT sublimate separator and charged to the reaction system together with the DMT sublimate that is in solution or in suspension, the glycol used as barrier liquid has a temperature of 20-160°C, preferably 110-130°C, and the volatile fractions entrained with the inert gas stream are precipitated in a water separator.

2. A method as in Claim 1, characterized by the fact that the inert gas stream introduced into the DMT sublimate separator is used at the same time for befogging the DMT and glycol systems.

3. A method as in Claims 1 and 2, characterized by the fact that the fresh glycol pumped through the separator has a temperature of 70-120°C.

4. A device for conducting the method as in Claims 1-3, characterized by the fact that the DMT sublimate separator (3) consists of three chambers (4, 5, 6), where the chamber (5) is provided with a partially heated ventilation line (13) and a connected water separator (9), chamber (4) is provided with a /7 glycol dispensing line (12) with distribution plate (7), an inert gas dispensing line (11) and heated DMT sublimate line (10), and chamber (6) is provided with a mixture outlet line (14).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